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6. AUTHOR(S) Professor B. M. Hillberry			
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13. ABSTRACT (Maximum 200 words) This instrumentation is a unique combination of an Environmental Scanning Electron Microscope (ESEM) with an electrohydraulic fatigue loading stage. The ESEM uses a patented technology that provides direct image capability in an environmental chamber that can contain gas or water vapor and in which temperature and pressure (to 50 Ton) can be varied. The computer controlled electrohydraulic loading stage can be operated in load, displacement or strain control. Resistance heating is used to control the specimen temperature (room temperature to 1150C). The combined ESEM and fatigue loading stage will provide direct on-line images (1 micron resolution) and quantitative data from test specimens under cyclic load (0 to 10 kN at 0 to 20 Hz). This unique system provides direct micro-level observational and loading conditions. It is currently being used to study the micromechanisms of fatigue crack formation and fatigue damage development of existing and advanced materials. This system makes it possible to study materials systems in ways that have not previously been possible.			
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**Air Force Office of Scientific Research
DURIP/95 Grant No. F49620-95-1-0507**

**Environmental Scanning Electron Microscope
with
Electrohydraulic Fatigue Loading Stage**

Principal Investigator: Professor B. M. Hillberry
School of Mecanical Engineering
Purdue University
14 May 1997

FINAL TECHNICAL REPORT

Equipment Purchase and Delivery

The Environmental Scanning Electron Microscope(ESEM manufactured by ElectroScan) with Electrohydraulic Fatigue Loading Stage(manufactured by Instron) purchased under this grant was exactly the equipment requested in the original proposal. The vendor quotes for the equipment were obtained prior to writing the proposal and were a part of the proposal. When the grant was awarded, we immediately ordered the equipment as quoted. The vendors provided the equipment at the quoted price and the budget was met exactly. The Purdue University cost share of 1/3 was provided as per the proposal. A copy of the original Budget is attached to this report.

The ESEM was installed January 1996 and the Fatigue Loading Stage was installed January 1997. The Fatigue Stage required considerable special development work as we had pushed the vendor in the original quote to higher temperatures and performance which resulted in the delivery date to be about 10 months late. Prior to delivery of the Fatigue Stage, it was shipped to the ElectroScan factory for integration with an identical ESEM and for performance evaluation. The Principal Investigator visited the factory at the time and approved the system for shipment to Purdue. Personnel from both ElectroScan and Instron participated in the installation of the Fatigue System at Purdue.

Equipment Description

The Environmental Scanning Electron Microscope uses a patented method of detecting the secondary electrons to create the surface image of the material being examined. This unique imaging technology allows direct observation of non-metallic materials without applying the conventional conductive coating to the sample. This is particularly important in studying polymers, ceramics and biological materials. In addition, the microscope does not require a high vacuum as do conventional SEM's. The system can operate with chamber pressures to 50 Torr, which allows studies in gaseous and moist environments to be conducted within the chamber.

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The Fatigue Loading Stage consists of a 2,000 pound load actuator that can be operated to 20 Hz frequency. In addition, a heating stage is included that can heat the specimen to 1150 degrees C during fatigue cycling or static loading. These capabilities exceed previous systems by twice the load capacity and frequency and 150 degrees higher temperature. Vibration has been minimized and under cyclic loading at lower frequencies(0.1Hz) the specimen can be observed at magnifications of 2500X and above, which is at the micron level.

System Utilization

The grant was awarded to the School of Mechanical Engineering, however the system has been installed in a separate room in the Electron Microscope Center of the School of Materials Engineering. This Center operates as a cost center for the electron microscopes and provides user training. Installing the ESEM in this location makes it available on a fee basis to everyone on campus. The fatigue stage is easily removed from the system so that the ESEM can be used when the fatigue stage is not in use. In addition the fatigue stage will be available for others to use on a more limited basis. The School of Materials Engineering provides management, training and maintenance for the ESEM. The Principal Investigator retains a priority in the use of the equipment. A training short course for graduate students has already been completed and there are currently two to three users of the ESEM daily.

The fatigue stage was installed in January 1997. At installation it met the primary performance specifications for load capacity, frequency and temperature. There have been some other design problems that are currently being corrected by the vendor under warranty. These include specimen grips and alignment and a few other minor items.

System Operation

This system will be used to study the behavior of a number of materials systems in ways that have not previously been possible. Among the first investigations will be in situ studies of the micromechanisms of fatigue crack formation and damage development in existing and advanced material systems when subjected to conventional and severe temperature and environmental conditions. We have some preliminary results with the systems that shows fatigue cracks forming and growing in and aluminum alloy(a typical aging aircraft material of interest) at magnifications of 2500X. This shows how the crack opens and closes. This opening and closing can readily be measured on the digital image with just a couple of clicks of the computer mouse. Previously it had been thought that in this material the crack grew on each load cycle, but our observations show that the crack halted for a number of cycles and then suddenly broke the barrier and there was a large growth in just one cycle. These studies will provide direct observations and quantitative data on the micromechanisms of the fatigue process under different environmental conditions, which will significantly enhance our understanding of the fatigue process.

Budget

Project Period
7 June 1995 to 6 June 1996

Environmental Scanning Electron Microscope (ESEM) ElectroScan Corporation

<u>Item</u>	<u>Description</u>	<u>Price</u>
1	Scanning Electron Microscope Model 2020 with LaB6 gun	\$ 256,000
2	Specimen Temperature Control Stage (Peltier).	7,900
3	Solid-State Backscattered Electron detector.	10,500
4	Micromanipulator/Microinjector.	10,000
5	Viewport	2,100
6	Accessory Rack	5,000
7	External Beam Control Interface.	2,700
8	ESEM Network Connection.	5,000
9	Scikosha VP1500 Video Printer	1,995
10	EDX Long Working Assembly	1,500
11	Water Chiller/Recirculator for ESEM (Neslab CFT-33)	2,125
12	Water Bath/circulator for ESEM Accessories (Caron 2050).	<u>1,795</u>
Subtotal		\$315,615

Fatigue Loading Stage Instron Corporation

<u>Item</u>	<u>Description</u>	<u>Price</u>
1	Fatigue Loading Stage with Heating System to 1150 C.	370,000
2	Non-recoverable engineering costs.	<u>40,000</u>
Subtotal		410,000
Total		725,615

University Cost Share (one-third) 241,872

Total Funds Requested from DOD \$483,743